

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Jeffrey E. Russel Examiner #: 62785 Date: 12-4-2002
Art Unit: 1654 Phone Number 30 8-3975 Serial Number: 09/815978
Mail Box and Bldg/Room Location: CMI-11D13/CMI 9807 Results Format Preferred (circle) PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need.

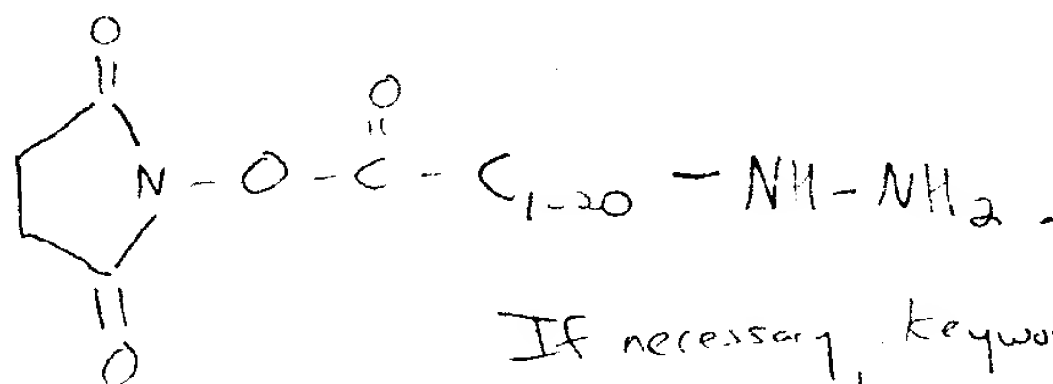
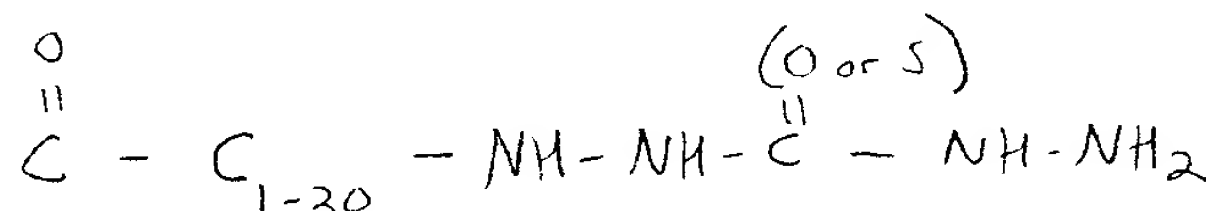
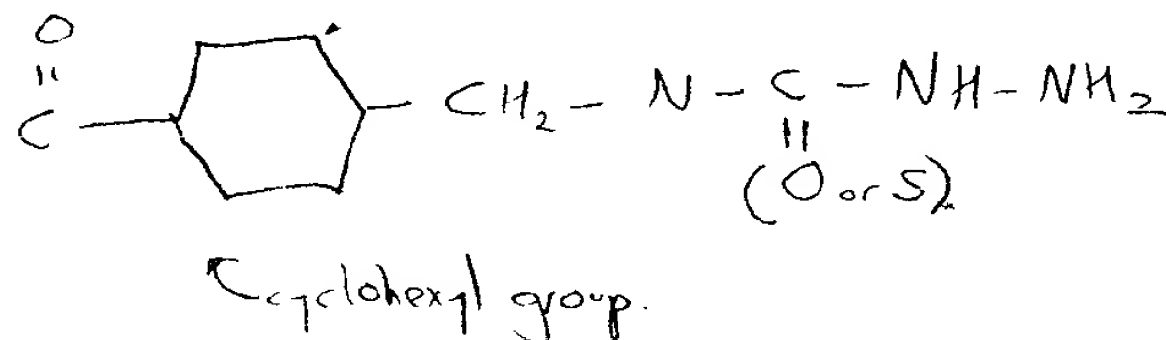
Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Hydrazine-Based And Carbonyl-Based Bifunctional Crosslinking Reagents
Inventors (please provide full names): D. Schwartz

Earliest Priority Filing Date: 3-22-2001

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Please search the following partial structure:



If necessary, keywords are conjugat?, crosslink?,
bifunctional, antibody, immobiliz?.

Edward Hart
Technical Info Specialist
STIC/Biotech
CMI 6B02 Tel: 305-9203

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DEC - 2002

Thank you.
JER

STAFF USE ONLY

Searcher: _____

Searcher Phone #: _____

Searcher Location: _____

Date Searcher Picked Up: 12/6/02

Date Completed: 12/12/02

Searcher Prep & Review Time: _____

Clerical Prep Time: _____

Online Time: _____

Type of Search

NA Sequence (#) _____

AA Sequence (#) _____

Structure (#) _____

Bibliographic _____

Litigation _____

Fulltext _____

Patent Family _____

Other _____

Vendors and cost where applicable

STN

Dialog

Questel/Orbit

Dr Link

Lexis/Nexis

Sequence Systems

WWW/Internet

Other (specify) _____

=> file hcaplus

FILE 'HCAPLUS' ENTERED AT 10:57:35 ON 12 DEC 2002

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FILE COVERS 1907 - 12 Dec 2002 VOL 137 ISS 24
FILE LAST UPDATED: 11 Dec 2002 (20021211/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

CAS roles have been modified effective December 16, 2001. Please check your SDI profiles to see if they need to be revised. For information on CAS roles, enter HELP ROLES at an arrow prompt or use the CAS Roles thesaurus (/RL field) in this file.

=> d stat que

L1 STR

12
31

2 7
C 3 C X C N N
1 C C 3 9 10 11

6 C C₄
C C C
14 13 5

VAR G1=O/S

NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 14

STEREO ATTRIBUTES: NONE

L3 135 SEA FILE=REGISTRY SSS FUL L1

L4 STR

9
G1

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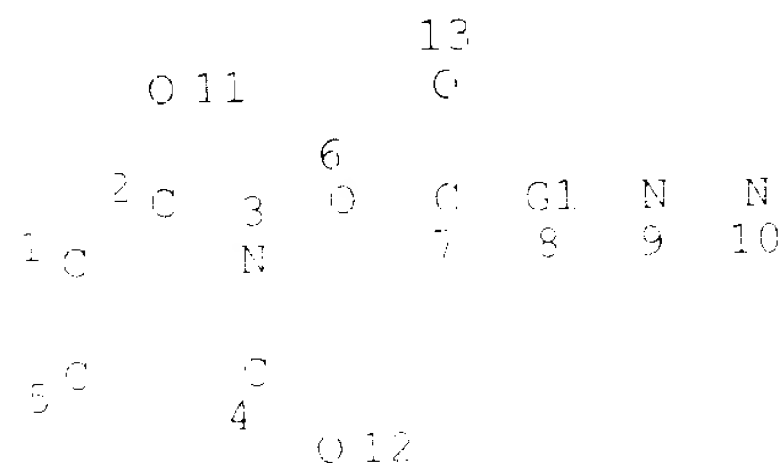
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REP G2=(1-20) C

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 DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
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 NUMBER OF NODES IS 9

STEREO ATTRIBUTES: NONE
 L6 78 SEA FILE=REGISTRY SSS FUL L4
 L9 STR



REF G1=(1-20) C
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 DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
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 NUMBER OF NODES IS 13

STEREO ATTRIBUTES: NONE
 L11 33 SEA FILE=REGISTRY SSS FUL L9
 L12 52 SEA FILE=HCAPLUS ABB=CN PLU=ON L3
 L13 37 SEA FILE=HCAPLUS ABB=CN PLU=ON L6
 L14 44 SEA FILE=HCAPLUS ABB=CN PLU=ON L11
 L16 3 SEA FILE=HCAPLUS ABB=CN PLU=CN L12 AND (CONUGAT? OR CROSSLINK
 ? OR BIFUNCTIONAL? OR ANITBODY? OR AB# OR MAB# OR PAB# OR
 IMMOBILI?)
 L17 4 SEA FILE=HCAPLUS ABB=CN PLU=ON L13 AND (CONUGAT? OR CROSSLINK
 ? OR BIFUNCTIONAL? OR ANITBODY? OR AB# OR MAB# OR PAB# OR
 IMMOBILI?)
 L18 3 SEA FILE=HCAPLUS ABB=CN PLU=ON L14 AND (CONUGAT? OR CROSSLINK
 ? OR BIFUNCTIONAL? OR ANITBODY? OR AB# OR MAB# OR PAB# OR
 IMMOBILI?)
 L19 11 SEA FILE=HCAPLUS ABB=CN PLU=ON L16 OF L17 OF L18

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L19 ANSWER 1 OF 10 HCAPLUS COPYRIGHT 2002 ACS
 ACCESSION NUMBER: 2001:713305 HCAPLUS
 DOCUMENT NUMBER: 135:272864
 TITLE: Hydrazine-based and carbonyl-based
bifunctional crosslinking reagents
 for biomolecules, drugs, and synthetic polymers
 INVENTOR(S): Schwartz, David A.
 PATENT ASSIGNEE(S): Solulink, Inc., USA
 SOURCE: PCT Int. Appl., 97 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001070685	A2	20011927	WO 2001-009252	20011021
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BE, BF, BG, BH, BI, BJ, BK, BL, BM, BN, BO, BR, BS, BT, BU, BV, BW, BY, BZ, CA, CB, CC, CD, CE, CF, CG, CH, CI, CJ, CK, CL, CM, CN, CO, CP, CQ, CR, CS, CT, CU, CV, CW, CX, CY, CZ, DD, DE, DF, DG, DH, DI, DJ, DK, DL, DM, DN, DO, DP, DQ, DR, DS, DT, DU, DV, DW, DX, DY, DZ, EA, EB, EC, ED, EE, EF, EG, EH, EI, EJ, EK, EL, EM, EN, EO, EP, EQ, ER, ES, ET, EU, EV, EW, EX, EY, EZ, FA, FB, FC, FD, FE, FF, FG, FH, FI, FJ, FK, FL, FM, FN, FO, FP, FQ, FR, FS, FT, FU, FV, FW, FX, FY, FZ, GA, GB, GC, GD, GE, GF, GH, GI, GJ, GK, GL, GM, GN, GO, GP, GQ, GR, GS, GT, GU, GV, GW, GX, GY, GZ, HA, HB, HC, HD, HE, HF, HG, HH, HI, HJ, HK, HL, HM, HN, HO, HP, HQ, HR, HS, HT, HU, HV, HW, HX, HY, HZ, IA, IB, IC, ID, IE, IF, IG, IH, II, IJ, IK, IL, IM, IN, IO, IP, IQ, IR, IS, IT, IU, IV, IW, IX, IY, IZ, JA, JB, JC, JD, JE, JF, JG, JH, JI, JJ, JK, JL, JM, JN, JO, JP, JQ, JR, JS, JT, JU, JV, JW, JX, JY, JZ, KA, KB, KC, KD, KE, KF, KG, KH, KI, KJ, KK, KL, KM, KN, KO, KP, KQ, KR, KS, KT, KU, KV, KW, KX, KY, KZ, LA, LB, LC, LD, LE, LF, LG, LH, LI, LJ, LK, LL, LM, LN, LO, LP, LQ, LR, LS, LT, LU, LV, LW, LX, LY, LZ, MA, MB, MC, MD, ME, MF, MG, MH, MI, MJ, MK, ML, MM, MN, MO, MP, MQ, MR, MS, MT, MU, MV, MW, MX, MY, MZ, NA, NB, NC, ND, NE, NF, NG, NH, NI, NJ, NK, NL, NM, NN, NO, NP, NQ, NR, NS, NT, NU, NV, NW, NX, NY, NZ, OA, OB, OC, OD, OE, OF, OG, OH, OI, OJ, OK, OL, OM, ON, OO, OP, OQ, OR, OS, OT, OU, OV, OW, OX, OY, OZ, PA, PB, PC, PD, PE, PF, PG, PH, PI, PJ, PK, PL, PM, PN, PO, PP, PQ, PR, PS, PT, PU, PV, PW, PX, PY, PZ, QA, QB, QC, QD, QE, QF, QG, QH, QI, QJ, QK, QL, QM, QN, QO, QP, QQ, QR, QS, QT, QU, QV, QW, QX, QY, QZ, RA, RB, RC, RD, RE, RF, RG, RH, RI, RJ, RK, RL, RM, RN, RO, RP, RQ, RR, RS, RT, RU, RV, RW, RX, RY, RZ, SA, SB, SC, SD, SE, SF, SG, SH, SI, SJ, SK, SL, SM, SN, SO, SP, SQ, SR, SS, ST, SU, SV, SW, SX, SY, SZ, TA, TB, TC, TD, TE, TF, TG, TH, TI, TJ, TK, TL, TM, TN, TO, TP, TQ, TR, TS, TT, TU, TV, TW, TX, TY, TZ, UA, UB, UC, UD, UE, UF, UG, UH, UI, UJ, UK, UL, UM, UN, UO, UP, UQ, UR, US, UT, UV, UW, UX, UY, UZ, VA, VB, VC, VD, VE, VF, VG, VH, VI, VJ, VK, VL, VM, VN, VO, VP, VQ, VR, VS, VT, VU, VW, VX, VY, VZ, WA, WB, WC, WD, WE, WF, WG, WH, WI, WJ, WK, WL, WM, WN, WO, WP, WQ, WR, WS, WT, WU, WV, WW, WX, WY, WZ, XA, XB, XC, XD, XE, XF, XG, XH, XI, XJ, XK, XL, XM, XN, XO, XP, XQ, XR, XS, XT, XU, XV, XW, XX, XY, XZ, YA, YB, YC, YD, YE, YF, YG, YH, YI, YJ, YK, YL, YM, YN, YO, YP, YQ, YR, YS, YT, YU, YV, YW, YX, YY, YZ, ZA, ZB, ZC, ZD, ZE, ZF, ZG, ZH, ZI, ZJ, ZK, ZL, ZM, ZN, ZO, ZP, ZQ, ZR, ZS, ZT, ZU, ZV, ZW, ZX, ZY, ZZ			
US 2002146504	A1	20021010	US 2002-00277	20020010
PRIORITY APPLN. INFO.:			US 2000-191186P	P 20000322
			US 2001-262094F	P 20010116

OTHER SOURCE(S): MARPAT 100-272864

[illegible]

362522-51-8P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent);
 (crosslinking agent; preparation of hydrazine- and carbonyl-based
 bifunctional crosslinking agents and use with
 bioms., drugs, and synthetic polymers)

L19 ANSWER 2 OF 10 HCAPLUS COPYRIGHT 2002 ACS
ACCESSION NUMBER: 1993:617414 HCAPLUS
DOCUMENT NUMBER: 119:217414
TITLE: Peptide aldehyde analogs for trypsin inhibitors
INVENTOR(S): Brunck, Terence Kevin; Pepe, Michael Gary; Fearsall,
Daniel Andrew; Webb, Thomas Roy
PATENT ASSIGNEE(S): Corvas International, Inc., USA
SOURCE: ECT Int. Appl., 01 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
NO 9814779	A1	19980805	NO 1998-US906	19980129
W: CA, JP				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				

EP 627925 A1 19941214 EP 1993-200110 1993-12-10
 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, NL, PT, SE, SI, TR
 JP 61503715 TE 19930420 JP 1993-11997 1993-11-24
 US 5494418 A 19960709 US 1993-11997 1993-11-24
 PRIORITY APPLN. INFO.:
 US 1992-027800 1992-11-17
 US 1993-11066 1993-11-24
 WO 1993-US906 1993-11-24

OTHER SOURCE(S): MARPAT 119:217.14

AB Peptide aldehyde analogs are disclosed which have substantial potency and specificity as inhibitors of mammalian pancreatic trypsin. The compds. of the invention are useful in the prevention and treatment of tissue damage or destruction associated with pancreatitis. Preparation of the analogs is described. Thus, N-t-butoxycarbonyl-L-Asp-L-Pro-L-argininal (I) (preparation given) had a K_i against trypsin of 0.00045 μ M. The effectiveness of I in an animal model for pancreatitis was also demonstrated.

IT 139976-30-0P

RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation and immobilization of, in peptide aldehyde analog preparation for trypsin inhibitor)

IT 139976-26-4P 139976-27-5P 139976-29-7P

139976-30-ODP, solid phase-immobilized

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RCT (Reactant or reagent)
 (preparation and reaction of, in peptide aldehyde analog preparation for trypsin inhibitor)

L19 ANSWER 3 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1992:611932 HCAPLUS

DOCUMENT NUMBER: 117:212932

TITLE: Total synthesis and absolute configuration of bengamide A

AUTHOR(S): Chida, Noritaka; Tobe, Takahiko; Okada, Shinsuke; Ogawa, Seishiro

CORPORATE SOURCE: Fac. Sci. Technol., Keio Univ., Yokohama, 223, Japan

SOURCE: Journal of the Chemical Society, Chemical Communications (1992), (15), 1064-6

CIDEN: JCCCAT; ISSN: 0022-4936

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 117:212932

GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB The first total synthesis of the novel marine natural product, bengamide A (I) is described, revealing the **absolute** configuration of this compound. I was prepared in several steps from known ester II (R¹ = H-3,4), which can be obtained from L-glutamic acid in 4 steps. Key steps were the cyclization of active ester III to give hexahydro-2-azepinone IV (R¹ = CH₂Ph, R² = Boc) and the coupling of IV.CF₃CO₂H (R¹ = R² = H) with polyhydroxylated C11 side chain V by (EtO)₂P(O)CN to give the corresponding amide.

IT 144090-64-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RCT (Reactant or reagent)
 (preparation and cyclization of)

L19 ANSWER 4 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1991:425407 HCAPLUS

DOCUMENT NUMBER: 115:25407
 TITLE: Novel trifunctional carrier molecule for the fluorescent labeling of haptens
 AUTHOR(S): Bredenist, Reinhard; Werhoff, Gregory A.; Kuntzsch, Anne M.; Charles, Paul T.; Thompson, Richard K.; Lygler, Frances S.; Vogel, Carl Wilhelm
 CORPORATE SOURCE: Dep. Biochem. Mol. Biol., Georgetown Univ., Washington, DC, 20007, USA
 SOURCE: Analytical Biochemistry (1991), 195:2, 272-277
 CODEN: ANBDA2; ISSN: 0003-2697
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 AB The authors developed a novel trifunctional carrier mol. for the synthesis of hapten-fluorophore conjugates as reporter mols. in immunoassays. This carrier eliminates some of the disadvantages associated with currently used fluorophore-labeling procedures including high nonspecific binding. The backbone of the carrier consists of the 21 amino acid residues of the insulin A-chain mol. This polypeptide provides a single site (terminal amino group) for covalent coupling of the hapten, three carboxyl groups for the attachment of fluorophores, and four sulfhydryl groups for derivatization with hydrophilic residues to compensate for the hydrophobic effect of the attached fluorophores. The sites for fluorophore attachment are 4, 17, and 21 amino acids away from the hapten attachment site. This spatial separation minimizes quenching of the fluorescence signal due to interaction of the fluorophores with each other and with the attached hapten. 2,4-Dinitrophenol (DNP) was selected as model hapten, fluorescein as label, and S-sulfonate groups as hydrophilic residues. The properties of the DNP-insulin A-chain-fluorescein conjugate (DNP-Ins-Fl) were compared to those of a DNP derivative labeled with a single fluorescein moiety via a small lysine spacer (DNP-Lys-Fl). The DNP-Ins-Fl conjugate exhibited a 3-fold lower nonspecific adsorption to **immobilized** non-immune Ig compared to an approx. 3-fold more efficient displacement from the binding sites of an **immobilized** anti-DNP antibody by the antigen DNP-lysine. Furthermore, at equimolar concentrations, the DNP-Ins-Fl generated a 2.6-fold higher fluorescent signal than DNP-Lys-Fl. Due to these properties of DNP-Ins-Fl, DNP-lysine could be detected with an approx. 10-fold higher sensitivity compared to DNP-Lys-Fl as labeled antigen. The use of DNP-Ins-Fl as reporter molecule in a competitive fluorimmunoassay allowed the quant. determination of picomole amounts of DNP-lysine.
 IT 134664-50-9
 RL: RCT (Reactant); FACT (Reactant or reagent)
 (reaction of, with FITC)

L19 ANSWER 5 OF 10 HCAPLUS COPYRIGHT 2002 ACS
 ACCESSION NUMBER: 1990:4 2:71 HCAPLUS
 DOCUMENT NUMBER: 115:2541
 TITLE: Preparation and characterization of immunoconjugates for antibody-targeted photolysis
 AUTHOR(S): Fikestraw, Scott L.; Tompkins, Ronald G.; Yarnack, Martin L.
 CORPORATE SOURCE: Cent. Adv. Biotech. Med., Rutgers, State Univ., Piscataway, NJ, 08855, USA
 SOURCE: Biotechnology Chemistry (1990), 1:1, 212-221
 CODEN: BICRE3; ISSN: 1043-1802
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 AB Monoclonal antibody (MAb)-dextran-tin(IV) chlorin e6(SnCe6) immunoconjugates were prepared by a new technique involving the use of reducing terminal-modified dextran carriers and site-specific modification of the Fc oligosaccharide moiety on the antibodies. Dextran carriers were synthesized to increase the number of SnCe6 mols. attached to a MAb

The dextran carriers were coupled to the **MAB** via a similar, chain-terminal hydrazide group to prevent aggregation of **MABs**. Conjugates were prepared with anti-melanoma **MAB** 2.1 containing 18.9 SnCe6 mols. per **MAB**. Under neutral conditions, hydrolysis of the hydrazone bond between the **MAB** and the dextran carrier could be detected, and the hydrazone was not stabilized by reaction with NaCNBH_3 or NaBH_4 . Anal. of the purified immunconjugates showed that approx. 2 dextran carrier chains were attached to a **MAB** regardless of the number of SnCe6 mols. linked to a dextran carrier. Site-specific covalent attachment of the SnCe6-dextran chains to the **MAB** was confirmed by SDS-PAGE. HPLC anal. of the conjugates gave a single species eluting in the range of 200-240 kDa. As determined by a competitive inhibition RIA using viable SK-MEL-2 human malignant melanoma cells, the conjugates showed excellent retention of antigen-binding activity relative to unconjugated **MAB**.

IT 127381-73-1P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and hydrazone-dextran terminal hydrazide protection by)

L19 ANSWER 6 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1974:13710 HCAPLUS
DOCUMENT NUMBER: 87:1871
TITLE: Production of a foam material
INVENTOR(S): Utsomi, Naoshi; Nakamura, Tetsuro
PATENT ASSIGNEE(S): Unifika Co., Ltd.
SOURCE: Jpn. Tokkyo Koho, 2 pp.
COLLEN: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 47151944	B4	19721227	JP 1969-88917	19691106

AE (α-Acetylenylidene) carbonylhydrazide (I) [50883-75-5]
(CH₃COO(C≡C):NNHCONHNH₂), which generated nontoxic, odorless, nonflammable gas on decomposition was used as a blowing agent for manufacture of polymer foams.

Thus, 93 parts **ABS** copolymer [9003-56-9] was dry-blended with 1 part (I) and injection molded at die temperature 200 deg. at 40 mm/min. to give a foam having uniform small cells and an apparent sp.gr. 0.14 g/cm³.

IT 50883-75-5

RL: USES (Uses)
(blowing agents, for manufacture of polymer foams)

L19 ANSWER 7 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1967:75307 HCAPLUS
DOCUMENT NUMBER: 67:75307
TITLE: Preparation of terephthaloyl diisocyanate
AUTHOR(S): Meidlein, Richard; Bottler, Rainer
CORPORATE SOURCE: Univ. Marburg-Lahn, Marburg-Lahn, Ger.
SOURCE: Chem. Ber. (1967), 100(2), 698-700
COLLEN: CHBEAM

DOCUMENT TYPE: Journal
LANGUAGE: German

GI For diagram(s), see printed CA Issue.

AB p-C₆H₄(CONH₂)₂ (32.8 g.) in 200 cc. dry CCl₄ refluxed about 10 days with 152.4 g. (COCl)₂ gave 46.2 g. p-C₆H₄(CONCO)₂ (I), m.p. 111-112°. (2.7 g.) in 60 cc. tetrahydrofuran treated with cooling with 1.5 g. absolute MeOH and stirred 1 hr. at room temperature yielded 1.4 g. p-C₆H₄(CONHCO₂R)₂ (II) (R = Me). Similarly prepared were the esters with R, m.p. (decomposition), and yields given: Et, 111-112°, 84% yield;

238-9°, 95; MeOCH₂CH₂, 159-61°, 32; Ph, 178-9°, 67.
Similarly prepared were p-ClC₆H₄(CONHCOX)₂ (III) (X = SPH), m. 124-5° (decomposition), 89; III (X = CHCH₂CH(OEt)₂), 85, m. 227-8° (decomposition); from MeOCH₂CH₂OH; and III (X = NHNHPh), m. 141-2° (decomposition). I (3.64 g.) in 50 cc. dry tetrahydrofuran treated dropwise with stirring with 17.1 cc. 8.5% dry HN₃-C₆H₅ gave 4 g. III (X = NH₃), m. 234-5°. I (2.4 g.) in 50 cc. dry tetrahydrofuran treated with stirring and cooling with 1 equivalent 6.2% CH₃N₃-Ph₃ gave 1.5 g. III (X = MeOCH₂CH₂OH).

IT 13506-12-2P 14994-19-5P

RI: SPN (Synthetic preparation); PSE (Preparation, preparation of)

L19 ANSWER 8 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1967:5172- HCAPLUS

DOCUMENT NUMBER: 60:557L-

TITLE: Hyarazine compounds as heteroconstituents in peptides.
VII. Synthesis of derivatives and peptides of
DL-α-hydrizine-β-phenylpropionic acid
(NHPhe).

AUTHOR(S): Gruppe, Renate; Medrich, Hartmut

CORPORATE SOURCE: Deut. Acad. Wiss., Berlin, Ger.

SOURCE: Chem. Ber., 1966, 99(12), 3914-24

CIEM: CHBEAM

DOCUMENT TYPE: Journal

LANGUAGE: German

AB of. CA 61, 307c. The following abbreviations are used: NHPhe =

α-hydrizine-β-phenylpropionic acid or -propionyl; NH₃ly =
hydrazinacetic acid or -acetyl; BOC = tert-BuO₂C; Z = PhCH₂CO₂C; OSu =
dicyclohexylcarbodiimide; Et = Et ester; OMe = Me ester; OSu =
hydroxysuccinimide ester; ONP = p-nitrophenyl ester; THF =
tetrahydrofuran; DMF = N,N-dimethylformamide. To 8.3 g. DL-NHPhe-OEt.HCl (CA 62, 1141) in 40 cc. **absolute** EtOH was added 5.4 cc. Et₃N and the product heated with 7.5 g. BOC-ONP 1 hrs. at 55° to give 1.5 g. II. DL-RNHNHCH(CH₂Ph)COX (I, R = BOC, X = OEt, III). Free NHPhe (2.44 g.) in 10 cc. EtOAc let stand 24 hrs. at room temperature with 1.57 g. BOC-N₃ gave 1.8 g. II. II (1.54 g.) in 20 cc. MeOH treated during 30 min. with 10 cc. N NaOH gave 1.3 g. I (R = BOC, X = OH, III). A solution of 1.80 g. NHPhe in 6 cc. 2N NaOH and 10 cc. dioxane stirred 24 hrs. at 35° with 1.57 g. BOC-N₃ and 0.05 g. MgO gave 1.3 g. III. II (1.54 g.) in 10 cc. saturated MeOH-NH₃ let stand 3 days, with 100 mg. 1,2,4-triazole gave 0.43 g. I (R = BOC, X = NH₂) (IV). To 1.54 g. II in 10 cc. **absolute** MeOH were added 100 mg. 1,2,4-triazole and 1 cc. 100% N₂H₄.H₂O and, after 2 days the MeOH was evaporated to give 1.35 g. I (R = BOC, X = N₂H₃) (V). To 33 g. PhCH₂OH in 125 cc. **absolute** C₆H₅N was added 61 g. ClCO₂C₆H₄NO₂-p at 0-5° with stirring and the reaction solution stirred 3 hrs. at room temperature to give 35.3 g. Z-ONP. Free NHPhe-OEt from 2.08 g. HCl salt treated with 2.73 g. Z-ONP like II (BOC-ONP procedure) gave 3.2 g. crude I (R = Z, X = OEt) (VI, decomposing on distillation. Crude VI (1.71 g.) saponified like

III (MeOH-aqueous NaOH method) gave 1.35 g. I (R = Z, X = OH, VII, m. 141-2°). 1.5 g. in 12.5 cc. 2N NaOH treated during 4 hrs. with 1.57 g. BOC-N₃ and 12.5 cc. 2N NaOH with ice cooling (the pH was kept at 11-12) and the mixture stirred 30 min. gave 4.3 g. III. VI (1 g.) treated with 10 cc. MeOH-NH₃ and 1,2,4-triazole like IV gave 1.3 g. I (R = Z, X = NH₂). VI (1.1 g.) treated like V gave 1.3 g. I (R = Z, X = N₂H₃). Free DL-NHPhe (1.03 g. HCl salt) kept 30 min. in 15 cc. Me₂CO gave 2.6 g. DL-MeC:NNHCH(CH₂Ph)COEt. III (1.4 g.) in 12 cc. DMF treated with 0.58 g. N-hydroxysuccinimide VIII, and then at 0° with 1.03 g. DCCl and the reaction mixture kept 60 hrs. at 0° gave 1.5 g. I (R = BOC, X = OSu (VIIIa). VII (3.14 g.) and 1.15 g. VIII in 15 cc. THF treated with 2.06 g. DCCl 24 hrs. at 0° gave 0.4 g. I (R = Z, X = OSu). DL-NHPhe-OEt.HCl (1.83 g.) suspended in 14 cc. THF, treated with 1.73 g.

Et₃N with ice cooling, the product treated with 1.5 g. Z-Gly and then with 1.52 g. DCCl with ice cooling, and the mixture kept overnight at 0° gave 1.66 g. NB-(Z-Gly)-LHPhe-R (IX, R = OEt, X = H). From NHPhe-OEt (from 2 g. HCl salt) in 30 cc. EtOAc kept 24 hrs. at room temperature with 0.2 g. Z-Gly-OCH₂CON gave 9.1 g. X, m. 86-87°. X (2 g.) saponified like III gave 1.5 g. IX (R = OH). X (3.99 g.) in 10 cc. **absolute** MeOH kept 3 days with 2.5 cc. 100% N₂H₄.H₂O and approx. 100 mg. 1,2,4-triazole gave 2 g. IX (R = N₂H₃). Free DL-NHPhe-OEt (from 2.4 g. HCl salt) and 2.1 g. Z-L-Ala in 30 cc. MeCN treated portionwise with 2.46 g. DCCl 24 hrs. at 0°, 2 drops AcOH added, and the mixture let stand 2 hrs., gave 2.4 g. NB-(Z-L-Ala)-DL-NHPhe-R (XII) (R = OEt, XIII). Free NHPhe-OEt (2.09 g.) in 5 cc. CHCl₃ combined with 3.44 g. Z-L-Ala-ONP in 6 cc. CHCl₃, 0.1 cc. AcOH added, and the solution kept 48 hrs. at room temperature gave 3.7 g. XII. XII (1.03 g.) saponified like III (MeOH-aqueous

NaOH

method) gave 0.62 g. XI (R = OH). XII (1.03 g.) in 8.4 cc. **absolute** AcOH heated 40 min. at 45° with 4.6 cc. approx. 4N HBr-AcOH gave 0.77 g. NB-(L-Ala)-DL-NHPhe-OEt.HBr (XIII.HBr), m. 206-11°. XIII.HBr (0.4 g.) in 4 cc. DMF treated with 1.5 g. Et₃N and then with 1.7 g. Z-L-Asp-ONP in 4 cc. THF gave 1.5 g. NB-(Z-L-Asp-L-Ala)-DL-NHPhe-OEt. To 1.54 g. Z-Gly in 4 cc. THF was added 0.423 g. Et₃N at -10° to -12° until pH 7 was attained, followed during 10 min. by 0.1 g. ClCO₂Et, the solution stirred approx. 1 hr. at -5°, treated with a precooled solution of 2.0 g. X in 12.5 cc. THF at -10°, stirred 10 min. at -5°, and refrigerated 3 days at 0° to give 0.5 g. Z-Gly-NHMFCH(CH₂Ph)COX (XIV) (R = Z-Gly, X = OEt) (XV). To 2 g. X and 1.64 g. Z-Gly in 30 cc. MeCN was added 1.23 g. DCCl with stirring and ice cooling and the solution let stand 20 hrs. at 0°, to give 0.6 g. XV. To 3.59 g. X in 20 cc. **absolute** C₅H₅N were added simultaneously 2.21 g. p-tosyl chloride and 1.66 cc. Et₃N with ice cooling to give 4.3 g. XVI (R = p-tosyl, X = OEt) (XVI). XVI (1.2 g.) dissolved in 1 cc. **absolute** MeOH by heating, the solution cooled, treated with approx. 100 mg. 1,2,4-triazole and 0.76 cc. 100% N₂H₄.H₂O, and let stand 4 days at room temperature gave 0.1 g. XIV (R = p-tosyl, X = N₂H₃). The mixed anhydride from 1.0 g. Z-Gly and 1.8 g. ClCO₂Et treated with a precooled solution of 2.0 g. II in 25 cc. THF as described for XV gave 1.2 g. crude NB-BOC-(Z-Gly)-DL-NHPhe-OEt (XVII). NB-tert-butyloxycarbonyl-L-α-hydrazino-β-phenylpropionyl amino acid esters was prepared as follows: Method A. To 3 millimoles appropriate amino acid ester-HCl in 4 cc. DMF was added 0.7 cc. Et₃N with stirring and ice cooling, precipitated Et₃N.HCl filtered and washed with 1 cc. DMF, the filtrate added to a solution of 1.4 g. III in 10 cc. DMF, 1.26 g. DCCl added at 0° and the solution kept approx. 60 hrs. at 0° to give the corresponding heterodipeptide ester. Method B. A solution of 3 millimoles amino acid ester (prepared as in Method A) combined with a solution of 1.88 g. VIIIa in 10 cc. THF, and kept approx. 60 hrs. at 20-25° gave 90-100% corresponding heterodipeptide ester. Thus, with Gly-OEt, there was obtained 91% by method A) and 100% by method B) NB-BOC-GL-NHPhe-Gly-OEt (XVIII). From L-Leu-OMe was obtained 70% (method A) and 80% (method B) diastereoisomeric mixture of NB-BOC-GL-NHPhe-L-Leu-OMe. L-Ile-Gly-L-Leu-L-Met-NH₂ (Luebke, et al., CA 62, 4113e) 0.467 g., 1 millimole Et₃N, and 1 millimole VIIIa in 4 cc. DMF let stand 60 hrs. at 20-25° and diluted with H₂O gave 0.62 g. NB-BOC-GL-NHPhe-L-Ile-Gly-L-Leu-L-Met-NH₂. A solution of 3.3 millimoles L-Ile-OMe (prepared as in method A) combined with a solution of 1.26 g. XI (R = OH) in 6 cc. DMF, and treated further like method A gave 1.7 g. NB-(Z-L-Ala)-DL-NHPhe-L-OMe. XVIII (1.82 g.) in 20 cc. MeOH combined with a solution of 0.2 g. NaOH in 50 cc. H₂O, a solution of 1.1 g.

NaOH

in 50 cc. H₂O and 20 cc. MeOH added dropwise during 2 hrs. while maintaining the pH at 8-9 gave 1.1 g. NB-BOC-GL-NHPhe-Gly-LH. XIX (1.82 g.) in 20 cc. THF treated first with 1.1 g. Z-L-Asp-ONP and then with 0.52 g. DCCl overnight at 0°, gave 1.66 g.

N β -BDC-DL-NHPhe-Gly-OEt, m. 90-2°.

IT 14381-16-9P 14381-17-0P

RL: SPN (Synthetic preparation); PPEP (Preparation)
(preparation of)

119 ANSWER 9 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1965:438638 HCAPLUS

DOCUMENT NUMBER: 63:38638

ORIGINAL REFERENCE NO.: 63:6854h, 6855a

TITLE: Synthesis of 1,3-bis[bis(carboxymethyl)amino]thiourea

AUTHOR(S): Ermakova, M. I.; Podgornaya, I. V.; Kuznetsov, N. V.;
Postovskii, I. Ya.

CORPORATE SOURCE: Chem. Inst., Sverdlovsk

SOURCE: Zh. Organ. Khim. (1965), 1(5), 857-60

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB (MeO₂CHCH₂·2NNH₂·HCl treated with aqueous NaOH gave the free ester, b₈ 124-5°, n_D20D 1.4562, d₄ 1.1930 [p-nitrobenzylidene derivative m. 75-7°; hydrazone with p,N-bis(β-chloroethyl)aminobenzaldehyde m. 74-8°; picrate m. 115-8°]. This kept 3-4 hrs. in EtOH-NH₃ with 332 gave 48% (MeO₂CHCH₂·2NNHCSNH₄, m. 102-4°, which adjusted to pH 3 with HCl gave the free acid, m. 82-4°, unstable in storage. This heated in absolute EtOH 50 min. gave SC[NHC(CH₂CO₂-Me)₂]₂, m. 86-8°, which refluxed 1 hr. in 10% HCl gave 33% SC[NHN(CH₂CO₂H)₂]₂, decomposed 190-3°. The polarograms of the salts of this acid with 13 common metal ions were reported. This acid in weakly basic medium can complex many metals such as Fe, Co, Ni, Mn, Cr(IV), and Cd. The complex forming tendency is weaker in acid media.

IT 2215-00-1, Acetic acid, [thiocarbonyl]dihydrazinylylidenehydrazide-
(preparation and polarography of its metal complexes

IT 2509-12-8, Acetic acid, [thiocarbonyl]dihydrazinylylidenehydrazide-
tetramethyl ester
preparation of)

119 ANSWER 10 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1964:469149 HCAPLUS

DOCUMENT NUMBER: 61:69149

ORIGINAL REFERENCE NO.: 61:11999e-h, 11999a-h, 12000a-h, 12001a-h, 12002a-h

TITLE: Synthesis of nitrogen-containing heterocycles. XXV.
α-Chloro oximes. 2

AUTHOR(S): Dornow, Alfred; Marquardt, Hans Heinrich; Paucksch,
Heinrich

CORPORATE SOURCE: Tech. Hochschule, Hannover, Germany

SOURCE: Ber. (1964), 97(8), 2105-8

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. CA 55, 2660h, 8386J. α-Chloro oximes with R₁(SCH₃)₂ (I) gave 2-aminothiazole 3-oximes (II). The 4-Me derivs. of II with R₂ = p-Cl-C₆H₄ and 2-amino-4-hydroxymethyl- and 4-chloromethylthiazole. α-Chloro oximes with EtOCS₂K gave α-ethoxythiocarbonylthiazole oximes which cyclized to 2-mercaptothiazole 3-oxides. PhCHClCCl:NH₂ (III) 11.0 g. in 20 cc. EtOH refluxed 1 hr. with 2.8 g. I in 15 cc. EtOH yielded 3.3 g. IV (R = Ph), m. 181° (H₂O). p-ClC₆H₄CHClCCl:NH₂ (4.1 g.) in 30 cc. EtOH treated 2 days at room temperature with 2.8 g. I in 15 cc. EtOH yielded

4.4 g. IV (R = p-ClC₆H₄) (V), m. 208° (EtOH). IV (R = Ph) (1 g.) in 25 cc. 2N HCl heated 0.5 hr. on a water bath with 2 g. Zn dust yielded 0.6 g. 2-amino-4-methyl-5-phenylthiazole (VI), m. 163° (aqueous MeOH). V (2 g.) in 20 cc. (CH₂Cl)₂ treated at room temperature with 1.6 cc. AcCl yielded

2.4 g. 2-amino-3-acetoxy-4-methyl-5(p-chlorophenyl)thiazolium chloride, m. 130° (decomposition). V (2 g.) in 150 cc. (CH₂Cl)₂ refluxed 1 hr. with

1.6 g. AcCl gave 0.6 g. 2-amino-4-methyl-5-phenyl-1,3,4-thiazole, m. 266° (decomposition) (C₆H₆), and 0.4 g. 2-amino-4-hydroxymethyl-5-(p-chlorophenyl)thiazole, m. 190° (C₆H₆-EtOH). EtOCS₂K (3.2 g.) in 25 cc. EtOH added to 3.7 g. III in 25 cc. EtOH and poured after 1 hr. into 400 cc. H₂O yielded 4.1 g. 1-ethoxythiocarbonylthio-2-oximino-1-phenylpropane (VII), m. 125° (aqueous EtOH). VII (3.5 g.) and 20 cc. 2N NaOH heated 20 min. at 100° gave 2 g. 2-SH analog (VIII) of IV (R = Ph), m. 143° (MeOH). VIII (0.6 g.), 5 cc. HI (d. 1.90), and 0.3 g. red P refluxed 20 min. yielded 0.4 g. 2-SH analog of VI, m. 131° (MeOH). XXVI. Use of α -amino oximes in the preparation of imidazole 3-oxides. Alfred Lornow and Hans Heinrich Marquardt. Ibid. 2169-72. α -Amino oximes react with ClCO₂Et (I) and ClCH₂Et (II) on the NH₂ group to yield the corresponding urethans and thio-urethans, resp. The free carboxylic acids, obtained by alkaline saponification of the urethans and thio-urethans, eliminate CO₂ and COS, resp., to yield with cyclization imidazole 3-oxides. Me-1(NOH)PhCHNH₂ (1.1 g.) in 40 cc. C₆H₆ treated at room temperature with stirring with 0.5 g. I in 10 cc. C₆H₆ gave 0.8 g. EtO₂CNHCHPhCHNH₂ (III), m. 112° (petr. ether-C₆H₆). III (0.3 g.) in 10 cc. 2N NaOH refluxed gave 0.5 g. 2-hydroxy-4,5,5-trimethylimidazole 3-oxide, m. 237° (H₂O). EtO₂C (0.15 g.) in 1 cc. 6N HCl added to 3 g. C in 50 cc. H₂O, and the mixture saturated with H gave the hydrogenation catalyst. This was stored under MeOH. A PhCH:NH (8.2 g.) in 80 cc. **absolute** MeOH and 1 cc. 10% HCl-MeOH hydrogenated at room temperature over 1.5 g. catalyst yielded 8.1 g. AcPhCHNH₂.HCl (IV), m. 201° (decomposition). IV (9.3 g.) and 1 g. NH₂OH.HCl in 30 cc. H₂O treated rapidly with stirring with 16.5 g. Ac₂O in 40 cc. H₂O (heated to 100°) gave 10.1 g. PhCH(NHCH₃)CMe:NOAc, m. 167° (iso-PrOH), which in 80 cc. H₂O treated with 1.5 g. Na₂CO₃ in 15 cc. H₂O and extracted with CHCl₃ yielded 6.7 g. PhCH(NHCH₃)CMe:NOH (V), m. 74° (CHCl₃-petr. ether), 76° (MeOH). V (3.3 g.) in 160 cc. C₆H₆ treated slowly with stirring with 1.1 g. I in 20 cc. C₆H₆ yielded 1.1 g. EtO₂CNHCHPhCMe:NOH, m. 158° (C₆H₆-petr. ether), which heated 10 min. on a water bath with 10 cc. 2N NaOH gave 1.1 g. VI, m. 102° (MeOH). VI (0.6 g.) in 30 g. 80% AcOH refluxed 3 hrs. on a water bath with 4 g. Zn dust gave 3.4 g. 2-hydroxy-4-methyl-5-phenylimidazole, m. 285° (aqueous EtOH). V (3.28 g.) in 150 cc. C₆H₆ treated slowly with stirring with 1.24 g. II in 30 cc. C₆H₆, stirred 1 hr., filtered from the HCl salt, m. 218°, and evaporated, and the viscous, yellow residue heated 4 hrs. on a water bath with 10 cc. 2N NaOH yielded 2.1 g. 2-SH analog of VI, m. 201° (decomposition, aqueous MeOH). XXVII. 1,2,4-Triazines. 1. Preparation of some new s-triazole [3,2-c]-as-triazines. Alfred Lornow, Herbert Menzel, and Paul Mark. Ibid. 2173-3. SO₂NHCH₂CH₂ (I) with α -oxo acids gave 4-amino-3-oxo-3-toloxo-2,3,4,5-tetrahydro-as-triazines (II) which formed, via the corresponding MeS comp., with amines 3,4-diamino-4,5-dihydroas-triazines (III). III were converted readily with HCO₂H or Ac₂O into 3,7-dihydro-s-triazole [5,4-c]-as-triazines (IV). I (53 g.) in 500 cc. boiling H₂O treated slowly with 44 g. AcCO₂H and kept 3 hrs. at room temperature yielded 75 g. II (R = Me) (V, m. 180° H₂O). I (1.06 g.) in 50 cc. boiling H₂O with 1.5 g. HCO₂H gave 2.1 g. II (R = Ph) (VI), decomposition 241° (H₂O). V (1 g.) in 1 cc. boiling MeOH treated with 1 cc. Et₃NH and refluxed 0.5 hr. yielded 1.3 g. 4-PhCH:N analog of V, m. 204-6° (C₆H₆). V (1 g.) in 10 cc. C₆H₆ treated 3 hrs. with 1 cc. AcCl gave 0.8 g. di-Ac derivative of V, m. 162° (C₆H₆). V (3.1 g.) and 1.6 g. NaOH in 30 cc. H₂O stirred 6 hr. with 1.3 cc. MeI yielded 2.7 g. H₂NNHC(SMe):NM:CMCO₂H (VII), m. 145-7° (aqueous MeOH). VII (1.8 g.) in 70 cc. MeOH refluxed 5 hrs. gave 1.7 g. VIII (R = Me) (IX, m. 165° (MeOH). V (15.7 g.) and NaOMe from 2.3 g. Na and 100 cc. **absolute** MeOH refluxed 2.5 hr. with 6.5 g. MeI yielded 1.1 g. VI (10.5 g.) and NaOMe from 2.3 g. Na and 100 cc. **absolute** MeOH treated during 10 min. dropwise with 13 g. MeI, refluxed

0.5 hr., and kept 12 hrs. at 10° yielded 23 g. VIII (R = Ph, R1 = H), m. 136° (MeOH). IX (1 g.) and 10 cc. BuNH₂ refluxed 5 hrs. yielded 1.1 g. III (R = Me, R1 = Bu), m. 135-6° (MeOH). IX (1 g.) and 10 cc. BuNH₂ heated 5 hrs. at 130° gave 0.5 g. III (R = Me, R1 = Ph), m. 135-6° (MeOH). IX (1 g.) with 4 cc. EtCH₂NH₂ yielded similarly 1.3 g. III (R = Me, R1 = EtCH₂) (XII), m. 160° (aqueous MeOH). X (1.17 g.) and 15 cc. BuNH₂ refluxed 1 hr. gave 0.87 g. III (R = Bu, R1 = Bu) (XIII), m. 142° (MeOH). X (2.34 g.) and 1 cc. EtCH₂NH₂ refluxed 1 hr. yielded 1.37 g. III (R = Bu, R1 = EtCH₂) (XIV), m. 175° (MeOH). X (1 g.) and 5 cc. morpholine heated 2 hrs. at 110° yielded 0.9 g. 4-amino-3-morpholino-5-oxo-6-phenyl-4,5-dihydro-1,2,4-triazine, m. 163° (MeOH). X (4 g.) in 25 cc. EtNH₂ heated 4 hrs. at 150° yielded 1.3 g. III (R = R1 = Ph) (XIV), m. 211.5° (MeOH). IX (1 g.) and 1.5 g. 98% N₂H₄ in 30 cc. **abs** iso-PrOH refluxed 4 hrs. gave 0.85 g. III (R = Me, R1 = NH₂), m. 283-5° (MeOH). [EtSC(NH₂):NHNH₂]Br (100 g.) in 250 cc. H₂O treated 24 hrs. at room temperature with 35 cc. 80% N₂H₄.H₂O yielded 66 g. HN=C(NHNH₂)₂.HBr (XV), m. 159° (MeOH). XV (17 g.) in 100 cc. H₂O heated 15 min. at 90° with 4.1 g. Ac₂O₂H yielded 12.4 g. III (R = Me, R1 = H) (XVI), m. 145° (H₂O). XV (4.3 g.) in a little H₂O with 3.8 g. Et₂COH in MeOH heated briefly gave 2 g. III (R = Ph, R1 = H) (XVII), m. 159-60° (decomposition). XVI (1 g.) and 3 cc. 99% HCCO₂H refluxed 4 hrs. yielded 0.55 g. IV (R = Me, R1 = R2 = H), m. 250-1° (H₂O). XVI (1 g.) and 3 cc. Ac₂O refluxed 3 hrs. yielded 0.7 g. IV (R = R1 = Me, R2 = H), m. 200-3°. XI (0.5 g.) and 1 cc. HCCO₂H refluxed 2 min. yielded 0.5 g. IV (R = Me, R1 = EtCH₂, R2 = H), m. 192° (H₂O). XII (1 g.) in 10 cc. EtOH refluxed 48 hrs. gave 1.2 g. IV (R = Ph, R1 = Bu, R2 = H), m. 186° (MeOH). XII (0.5 g.) gave similarly 0.4 g. IV (R = R1 = Ph, R2 = H), m. 212° (iso-PrOH). XIII (0.5 g.) and 1 cc. HCCO₂H refluxed 3 hrs. yielded 0.4 g. IV (R = Ph, R1 = EtCH₂, R2 = H), m. 191-1° (MeOH). XVII (0.5 g.) yielded similarly with 5 cc. Ac₂O 0.15 g. IV (R = Ph, R1 = H, R2 = Me), m. 247-8°. XVI (1 g.) in 60 cc. MeOH refluxed with 1.4 g. EtCH₂NH₂ yielded 0.7 g. XVIII, m. 201-2° (HCCNMe₂). XVII (1 g.) and 5 cc. Ac₂O heated 1 hr. at 131° yielded 0.3 g. solid, C₁₂H₈N₄O₂, m. 214° (MeOH), presumably a pyrazolo-as-triazine, and 1 g. light yellow prisms, C₁₂H₁₇N₅O₂, m. 123°, a di-Ac compound XXVIII. 1,2,4-Triazines. 2. Preparation of some new s-triazolo[4,3-b]-astriazines. Alfred Dornow, Werner Abelle, and Herbert Menzel. Ibid. 1179-84. 3-Hydrazino-1,2,4-triazines with CS₂, urea, or HCCN yielded s-triazolo[4,3-b]-as-triazines. 3-Methylthio-5,6-diphenyl-as-triazine (10 g.) and 10 cc. 80% N₂H₄.H₂O in 200 cc. iso-PrOH refluxed 12 hrs. yielded 16 g. 3-hydrazino-5,6-diphenyl-as-triazine I, m. 170° (MeOH). I and aldehydes or ketones in EtOH refluxed 12 hrs. gave in most cases nearly quant. the 3,6-diphenyl-1,2,4-triazin-3-ylhydrazones of the following compds. (m.p. given): Ph₂CO, 223° (MeOH); Ph₂C=O, 231° (HCCNMe₂); p-ClC₆H₄CHO, 234° (HCCNMe₂); p-MeOC₆H₄CHO, 268° (HCCNMe₂); furfural, 233° (EtOH); Me₂CO, 198° (Me₂CO); AcPh, 100° (iso-PrOH); EtPh, 112° (iso-PrOH); cyclohexanone, 162° (iso-PrOH). 3-Methylthio-5-oxo-6-ethyl-4,5-dihydro-1,2,4-triazine (1.0 g.) in 60 cc. iso-PrOH refluxed 5 hrs. with 15 cc. EtNH₂.H₂O gave MeNH and 13.6 g. 5-oxo-3-hydrazino-6-ethyl-4,5-dihydro-1,2,4-triazine III, m. 240°. III (1 g.) in 100 cc. boiling MeOH treated with 2.1 g. p-ClC₆H₄CHO yielded 1.1 g. 5-oxo-6-methyl-4,5-dihydro-1,2,4-triazin-3-ylhydrazone IV, m. 331° (HCCNMe₂). Similarly was prepared the analogous derivative of p-MeOC₆H₄CHO, m. 305° (HCCNMe₂), in 96% yield. IV (R = EtO, R1 = SH) (11 g.) and NaOEt from 1.5 g. Na and 210 cc. **absolute** EtOH treated 48 hrs. with 8 g. MeI gave 8.5 g. IV (R = EtO, R1 = MeS) (V), m. 143-5° (H₂O). IV (R = CH, R1 = MeS) (11 g.) and 15 cc. concentrated H₂SO₄ in 400 cc. **absolute** EtOH refluxed 5 hrs. yielded 8.2 g. V, m. 142° (H₂O). V (2 g.) in 100 cc. iso-PrOH refluxed 3 hrs. with 3 cc. 98% N₂H₄ yielded 1.4 g. IV (R = R1 = NHNH₂), did not melt up to 380° (aqueous MeOH). IV (R = CH, R1 = Ph) (1 g.)

and 6 g. ClCH₂CO₂H in 60 cc. H₂O refluxed 4 hrs. yielded 1.4 g. VII (R = OH, R' = OH, R'' = OH), m. 238° (H₂O). I (10 g.), 10 cc. MeOH, and 10 cc. C₅H₅N refluxed 10 hrs. yielded 10.5 g. VI (R = Ph, R' = OH, R'' = OH), m. 298-300° (HCONMe₂). I, C₅H₅N, and CS₂ deposited at room temperature a yellow precipitate, m. 100°, which heated in MeOH decomposed into its components. II (8 g.), 250 cc. C₅H₅N, and 10 cc. MeOH refluxed 10 hrs. and then heated about 60 hrs. on a water bath until the H₂O had evaporated, and yielded 5.2 g. VI (R = Me, R' = OH, R'' = OH), m. 298-300° (HCONMe₂). 4,5-Diamino-3-thioxo-2,3-dihydro-1,2,4-triazole (1.31 g.) in 60 cc. MeOH refluxed 5 hrs. with 0.45 g. AcCO₂H yielded 1.2 g. VIII, decomposition 305-310° (MeOH). VII (5 g.) in 500 cc. 5% aqueous K₂CO₃ treated 6 hrs. with 8 g. NaI gave 5 g. IX (R = Ph, R' = Me, R'' = Me), decomposition 197° (MeOH). VIII (3 g.) in 10 cc. 4% aqueous NaOH shaken 0.5 hr. with 1.5 cc. MeI and adjusted to pH 6 with AcOH yielded 2.8 g. IX (R = (R' = Me, R'' = OH) (X), m. 258-7° (MeOH). 4,5-Diamino-3-methylthio-1,2,4-triazole (1.45 g.) in 100 cc. H₂O refluxed about 3 hrs. with 0.95 g. AcCO₂H gave 1.51 g. X, m. 256-8° (MeOH). VII (2 g.) and 10 g. ClCH₂CO₂H in 100 cc. 70% AcOH refluxed 4 hrs. gave 1 g. IX (R = Ph, R' = Ph, R'' = CH₂CO₂H) (XII), m. 256° (AcOH); Me ester m. 161° (MeOH). VIII (4 g.) and 40 cc. 10% aqueous HCH₃CO₂H refluxed 1 hr. yielded 4.2 g. IX (R = Me, R' = OH, R'' = CH₂CO₂H), m. 256° (H₂O). XI (1 g.) in 50 cc. 10% aqueous KOH refluxed 6 hrs. gave 1 g. orange-red 5-hydroxy-6,7-diacenyl-8-triazole (4,5-diamino-1,2,4-triazine) (XII), decomposition 178-80° (H₂O). XI (1 g.) and 1 g. MeOH heated 10 min. at 220° yielded 0.4 g. XII, decomposition 275° (MeOH). I (1 g.) in 10 cc. concentrated HCl diluted with 50 cc. H₂O and filtered,

and

the residue dissolved in 10 cc. H₂O and treated dropwise with 1.2 g. NaOH in 10 cc. H₂O gave 7 g. 3-aminocarbazo-5,6-diacenyl-2,3-dihydro-as-triazine (XIII), decomposition 205-6° (H₂O). XIII (5 g.) heated 20 min. at 220° yielded 3 g. XII, decomposition 178-80° (MeOH). XXIX. 1,2,4-Triazines. 3. Alfred Dornow, Herbert Menzel, and Paul Marx. Ibid. 2185-6. The preparation of I, II (R = Me) (III), II (R = H) (IV), and V is described. 3-Methylthio-3-oxo-1,2,4-dimethyl-2,5-dihydro-1,2,4-triazine (10 g.) in 200 cc. absolute 2-propanol treated 48 hrs. at room temperature with 10 cc. 98% H₂SO₄ yielded 0.5 g. 5-oxo-3-hydrazino-2,6-dimethyl-2,5-dihydro-1,2,4-triazine (VI), m. 248° (HCONMe₂). VI (0.5 g.) in 400 cc. boiling MeOH treated with 0.4 g. Br₂ yielded 0.4 g. yellow 5-benzaldehydrazono analog of VI, m. 234° (MeOH). VI (0.5 g.) and 5 cc. HCO₂H refluxed 3 hrs. yielded 0.3 g. I, m. 131° (H₂O). VI (1.55 g.) in 10 cc. 2N HCl treated dropwise slowly with stirring with 5% aqueous NaNO₂ yielded 0.7 g. III, m. 101° (C₆H₆-petr. ether). 5-Oxo-3-hydrazino-6-phenyl-1,2,3,4-tetrahydro-1,2,4-triazine (VII) (6 g.) in 50 cc. 2N HCl with 3 g. NaNO₂ in 10 cc. H₂O gave 4g. IV, m. 214° (MeOH). 5-Hydrazinotetrazole (1 g.) in 10 cc. hot H₂O treated slowly with 0.86 g. AcOH gave 1.0 g. the 5-tetrazolylihydrazono (VIII), m. 215° (decomposition) (MeOH), of AcCO₂H. VIII (1 g.) and 5 cc. AcOH heated to solution and kept 14 hrs. yielded IV, m. 214° (H₂O), and some 5-acetamidotetrazole, m. 270° (decomposition) (AcOH). IV (1.1 g.) treated 12 hrs. at room temperature with 100 cc. C₆H₅NH₂SO₂ acid 10 g. H₂CONMeNO yielded 1.1 g. isomer of III, m. 210° (C₆H₆). VII (1.2 g.) in 50 cc. refluxing MeOH treated during 6 min. with 0.5 g. Br₂ and refluxed 3 hrs. gave 0.5 g. 6-tetrazolylacetophenone 1-oxo-4-methyl-1,4,5-dihydro-3,5-bis-1,2,4-triazin-3-ylhydrazono (IX), m. 194-5° (MeOH). VII (1 g.) in 50 cc. MeOH refluxed 1 hr. with 1.2 g. BrCH₂Me yielded 1.4 g. IX, m. 198° (MeOH). VII (3.5 g.) and 6 g. BrCH₂Br in 10 cc. HCONMe₂ heated 1 hrs. at 100° gave 5.2 g. 6-oxo-7-methyl-3-phenyl-5,9-dihydro-4H-as-triazino[4,3-b]-as-triazine, m. 303° (decomposition) (HCONMe₂). XXX. 1,2,4-Triazines. 4. Preparation of 1,3,4-thiadiazolo[2,3-c]-as-triazines. Alfred Dornow and Paul Marx. Ibid. (9), 2643-6. 3,4-Diamino-1,2,4-triazines and their 3-MeS analogs gave with CS₂ in C₅H₅N with the elimination of amine or MESH, resp., the pyridinyl salts I. II (R = Ph, R' = PhNH) (5.1 g.) in 30 cc. dry Et₂OH treated 1 hr. with 10 cc. CS₂ yielded 6.1 g. I (R = Ph) (III), m. 235° (H₂O). II

(R = Ph, R' = MeS) (5.0 g.), 50 cc. C₅H₅N, and 10 cc. CS₂ refluxed 12 hrs. and kept 12 hrs. at room temperature yielded 6.2 g. III. III (5.0 g.) in 50 cc. boiling H₂O adjusted with concentrated HCl to pH 1 yielded 3.9 g. compound IV (R = Ph) (V), m. 245° (decomposition) (1:1 HCONMe₂-MeOH). II (R = Me, R' = MeS) (5.0 g.), 40 cc. C₅H₅N, and 10 cc. CS₂ refluxed 3 hrs. yielded about 6 g. VI. VI (5.0 g.) in 50 cc. boiling H₂O adjusted with concentrated HCl to pH 1 gave 3.6 g. IV (R = Me) (VII), m. 240-1° (decomposition) (H₂O). V in aqueous NaOH heated briefly yielded 1.5 g. VIII (R = Ph) (IX), m. 244° (decomposition) (1:1 aqueous MeOH). 5-Hydrazono-2-thioxo-1,2,4-thiadiazolidine-HCl (X.HCl) in MeOH neutralized with aqueous Na₂CO₃ and treated with BrCO₂H gave quant. IX. VII dissolved in dilute aqueous NaOH and acidified gave quant. VIII (R = Me) (XI), m. 216-18° (H₂O). VII refluxed 1 hr. with dilute HCl gave 100% XI. X in hot H₂O treated dropwise with BrCO₂H gave quant. XI, m. 217-19° (H₂O). IX (2 g.) in 15 cc. AcOH refluxed 5 min. gave 2 g. XII (R = Ph, R' = Ac) (XIII), m. 215° (decomposition) (Ac₂O). XIII (1.00 g.) in 15 cc. MeOH refluxed 15 min. gave 1.41 g. V, m. 242°. V (1.5 g.) with 1.2 g. Na₂CO₃ in 7 cc. H₂O yielded 1.5 g. yellow XIV (R = Ph) (XV), m. 192° (decomposition) (H₂O). Similarly was prepared the pale yellow XIV (R = Me) (XVI), m. 205° (decomposition) (H₂O). XV (2.5 g.) in 250 cc. H₂O treated dropwise with stirring with 1.4 g. MeI and stirred 2 hrs. at room temperature gave 1.5 g. XII (R = Ph, R' = Me, m. 165° (C₆H₆-petr. ether). XVI (2.0 g.) and 1.7 g. MeI gave similarly 1.5 g. XII (R = R' = Me, m. 195-6° (H₂O). V (1.0 g.) and 150 cc. aqueous Na₂CO₃ heated to 30-40°, treated dropwise with 1.0 g. MeI, and heated 0.5 hr. on water bath gave 1.3 g. XVII (R = Ph, R' = Me, m. 218-19° (AcOH). VII (1.5 g.) in 20 cc. 4% aqueous NaOH shaken with 1.2 g. MeI, kept 1 hr., and adjusted with HCl to pH 3 gave 1.4 g. XVII (R = R' = Me, m. 217-18° (H₂O). XV (1.0 g.) in 10 cc. HCONMe₂ refluxed 5 min. with 0.3 g. PhCH₂Cl yielded 1.15 g. XII (R = Ph, R' = PhCH₂), m. 170° (C₆H₆-petr. ether). XVI (1.0 g.) in 6 cc. HCONMe₂ refluxed a few min. with 0.7 g. PhCH₂Cl yielded 1.25 g. XII (R = Me, R' = PhCH₂), m. 171° (MeOH). VII (1.0 g.) in 10 cc. 10% ClCH₂CO₂H refluxed 15 min. gave 1.3 g. XVII (R = Me, R' = CH₂CO₂H, m. 219-20° (decomposition) (H₂O). XV (1.0 g.) in 10 cc. HCONMe₂ refluxed briefly with 0.5 g. ClCH₂CO₂H gave 0.85 g. XII (R = Ph, R' = CH₂CO₂H (XVIII), m. 220° (MeOH). XVI (1.0 g.) and 0.5 g. ClCH₂CO₂H gave similarly 0.75 g. XII (R = Me, R' = CH₂CO₂H), m. 207° (H₂O). XVIII (1 g.) and 30% aqueous NaOH refluxed 5 hrs. and acidified yielded 1 g. XVII (R = Ph, R' = CH₂CO₂H), m. 197° (decomposition) (AcOH). XVI (1.0 g.) in 40 cc. H₂O treated dropwise with iodine in MeOH until the color persisted gave 0.8 g. XIX, m. 200° (decomposition).

IT 89715-26-4, Pyruvic acid, azine with S-Me thiocarbazate
(preparation of)

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<http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf>

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L20 ANSWER 1 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 362522-51-8 REGISTRY

CN Hydrazinecarbothioamide, N-[[trans-4-[[[2,5-dioxo-1-pyrrolidinyl]oxy]carbonyl]cyclohexyl]methyl]-, monohydrate
(CA INDEX NAME)

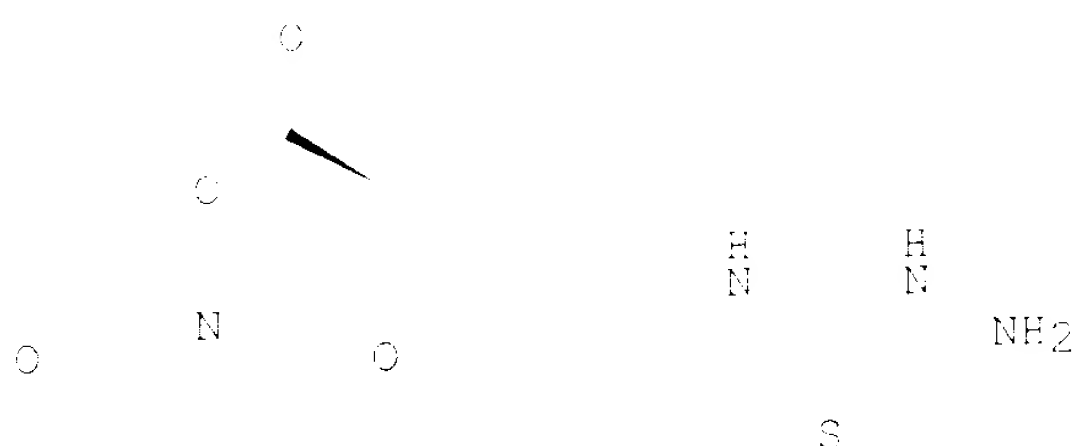
PS STEREOSEARCH

MF C13 H20 N4 O4 S . CL H

SR CA

LC STN Files: CA, CAPLUS, USPATFULL

Relative stereochemistry.



● HCl

1 REFERENCES IN FILE CA (1962 TO DATE)
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 135:272864

L20 ANSWER 2 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 144090-64-2 REGISTRY

CN Carbamic acid, [5-azido-1-[[[(2,5-dioxo-1-pyrrolidinyl)oxy]carbonyl]-4-(phenylmethoxy)pentyl]-, 1,1-dimethylethyl ester, [S-(R*,R*)]- (9CI) (CA INDEX NAME)

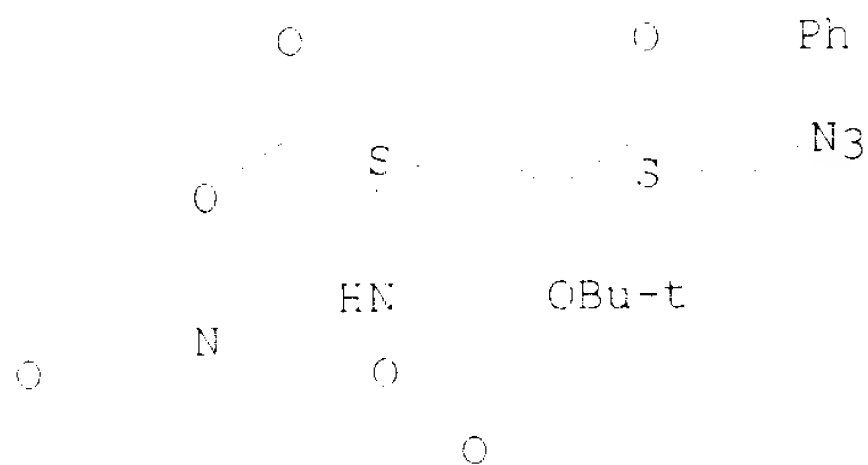
FS STEREOSEARCH

MF C22 H29 N5 O7

SR CA

LC STN Files: CA, CAPLUS, CASREACT

Absolute stereochemistry.



1 REFERENCES IN FILE CA (1962 TO DATE)
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 117:212932

L20 ANSWER 3 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 139976-30-0 REGISTRY

CN Cyclohexanecarboxylic acid, 4-[(7S)-7-[3-[[imino(nitroamino)methyl]amino]propyl]-11,11-dimethyl-3,9-dioxo-10-oxa-2,4,5,8-tetraazadodec-5-en-1-yl]-, trans- (9CI) (CA INDEX NAME)

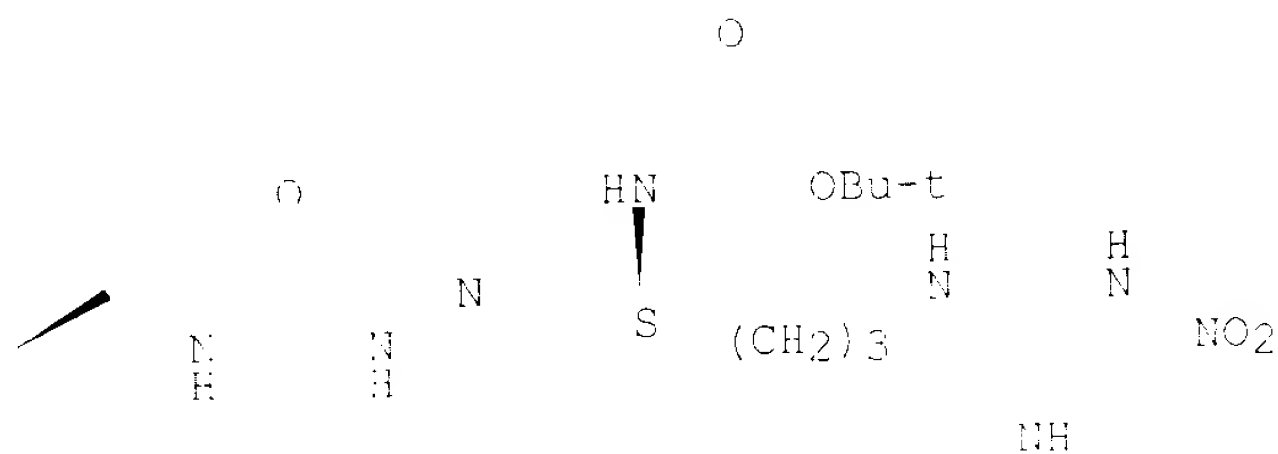
OTHER CA INDEX NAMES:

CN Cyclohexanecarboxylic acid, 4-[7-[3-[[imino(nitroamino)methyl]amino]propyl]-11,11-dimethyl-3,9-dioxo-10-oxa-2,4,5,8-tetraazadodec-5-en-1-yl]-, [4(S)-trans]-

RUSSEL 09 / 615976

FS STEREOSEARCH
MF 020 H36 NR 01
ER CA
DT STN Files: BRIDSTEIN*, CA, CAPERS, CRAFTSMAN
*File contains numerically sorted property data

Absolute stereochemistry.
Double bond geometry unknown.

 HO_2C

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

15 REFERENCES IN FILE CA (1962 TO DATE)
7 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
15 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE	1:	132:211478
REFERENCE	2:	131:229021
REFERENCE	3:	130:213549
REFERENCE	4:	127:220992
REFERENCE	5:	126:131740
REFERENCE	6:	125:196331
REFERENCE	7:	124:344120
REFERENCE	8:	124:176940
REFERENCE	9:	122:133851
REFERENCE	10:	121:212001

120 ANSWER 4 OF 16 REGISTRY COPYRIGHT 2002 ACS

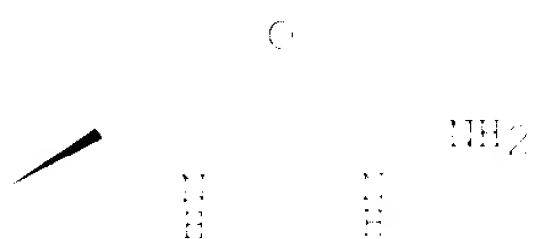
```

AN 139976-29-7  REGISTRY
CN Cyclohexanecarboxylic acid, 4-[[[(hydrazinocarbonyl)amino]methyl]-, trans-,
   mono(trifluoroacetate) (9CI)  (CA INDEX NAME)
FS SIERESEARCH
MF C6 H17 N3 O3 . C2 H F3 O2
BR CA
LC STN Files:  CA, CAPLUS, USPATFILL

```

CBN 139976-28-6
CMF C9 H17 N3 C3

Relative stereochemistry.



HO2C

CM 2

GRN 76-05-1
CMF C2 H F3 O2

F

F C CC2H

F

20 REFERENCES IN FILE CA (1962 TO DATE)
2 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
20 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 137:125292
REFERENCE 2: 137:125391
REFERENCE 3: 137:125390
REFERENCE 4: 137:109484
REFERENCE 5: 137:33541
REFERENCE 6: 134:281136
REFERENCE 7: 134:17726
REFERENCE 8: 133:17829
REFERENCE 9: 132:251428
REFERENCE 10: 130:223589

L20 ANSWER 5 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 139976-27-5 REGISTRY

CN Hydrazinecarboxylic acid, 2-[[[(trans-4-carboxycyclohexyl)methyl]amino]carbamoyl]-, 1-(1,1-dimethylethyl), ester [901] CA INDEX NAME

OTHER CA INDEX NAMES:

CN Hydrazinecarboxylic acid, 2-[[[(4-carboxycyclohexyl)methyl]amino]carbamoyl]-, 1-(1,1-dimethylethyl), ester, trans-

FS STEREOSEARCH

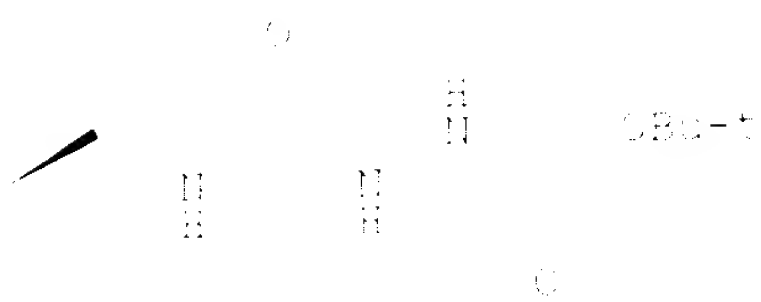
MF C14 H25 N3 O5

SR CA

LC STN Files: BEILSTEIN*, CA, CAPLUS, USPATFULL

File contains numerically searchable property data

Relative stereochemistry.



HO2C

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

21 REFERENCES IN FILE CA (1962 TO DATE)
21 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 137:3.541
REFERENCE 2: 134:2.1136
REFERENCE 3: 134:115970
REFERENCE 4: 133:232750
REFERENCE 5: 172:161428
REFERENCE 6: 151:329021
REFERENCE 7: 140:123539
REFERENCE 8: 128:205143
REFERENCE 9: 127:346661
REFERENCE 10: 127:120992

120 ANSWER 6 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 139976-26-4 REGISTRY

CN Hydrazinecarboxylic acid, 2-[[[trans-4-[(phenylmethoxy carbonyl)amino]ethyl]amino]carbonyl]-, 1,1-dimethylethyl ester (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Hydrazinecarboxylic acid, 2-[[[4-[(phenylmethoxy carbonyl)amino]ethyl]amino]carbonyl]-, 1,1-dimethylethyl ester, trans-

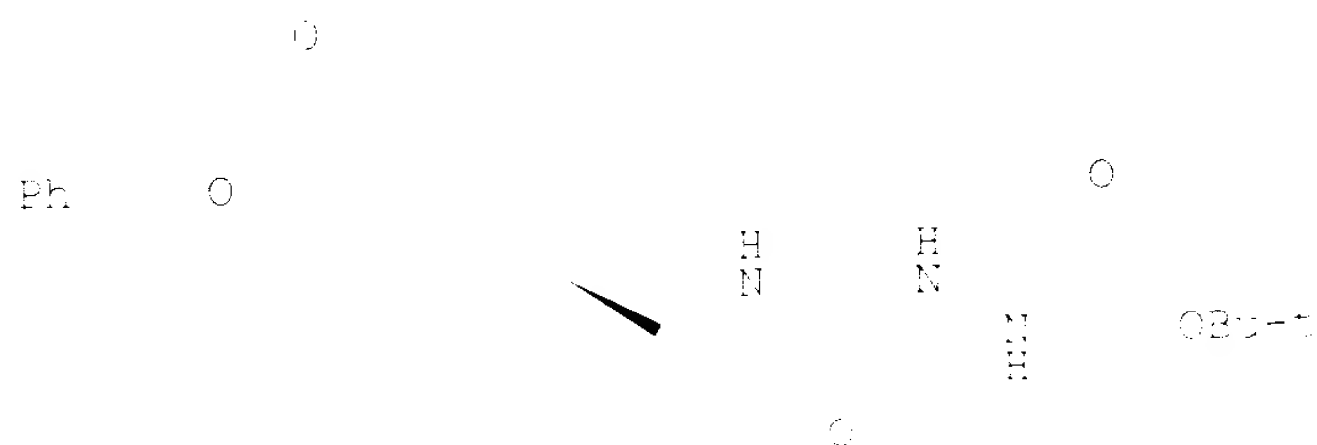
FS STEREOSEARCH

MF C21 H31 N3 O5

SR CA

LC STN Files: BILSTEIN*, CA, CAPLUS, USPATEFULL
(*File contains numerically searchable property data)

Relative stereochemistry.



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

19 REFERENCES IN FILE CA (1962 TO DATE)
19 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 132:251428
REFERENCE 2: 131:223031
REFERENCE 3: 130:223539
REFERENCE 4: 130:125401
REFERENCE 5: 129:205143
REFERENCE 6: 127:346661
REFERENCE 7: 127:220992
REFERENCE 8: 126:131713
REFERENCE 9: 125:196333
REFERENCE 10: 124:344120

L20 ANSWER 7 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 134664-50-9 REGISTRY

CN Insulin (cattle-A reduced), N-(2,4-dinitrophenyl)-, tris[2-(hydrazinocarbonyl)hydrazide], 6,7,11,20-tetrakis(hydrogen sulfate) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Insulin (ox-A reduced), N-(2,4-dinitrophenyl)-, tris[2-(hydrazinocarbonyl)hydrazide], 6,7,11,20-tetrakis(hydrogen sulfate)

FS PROTEIN SEQUENCE; STEREOSEARCH

MF C106 H165 N39 O50 S8

SR CA

LC STN Files: CA, CAPLUS

Absolute stereochemistry.

PAGE 1-A

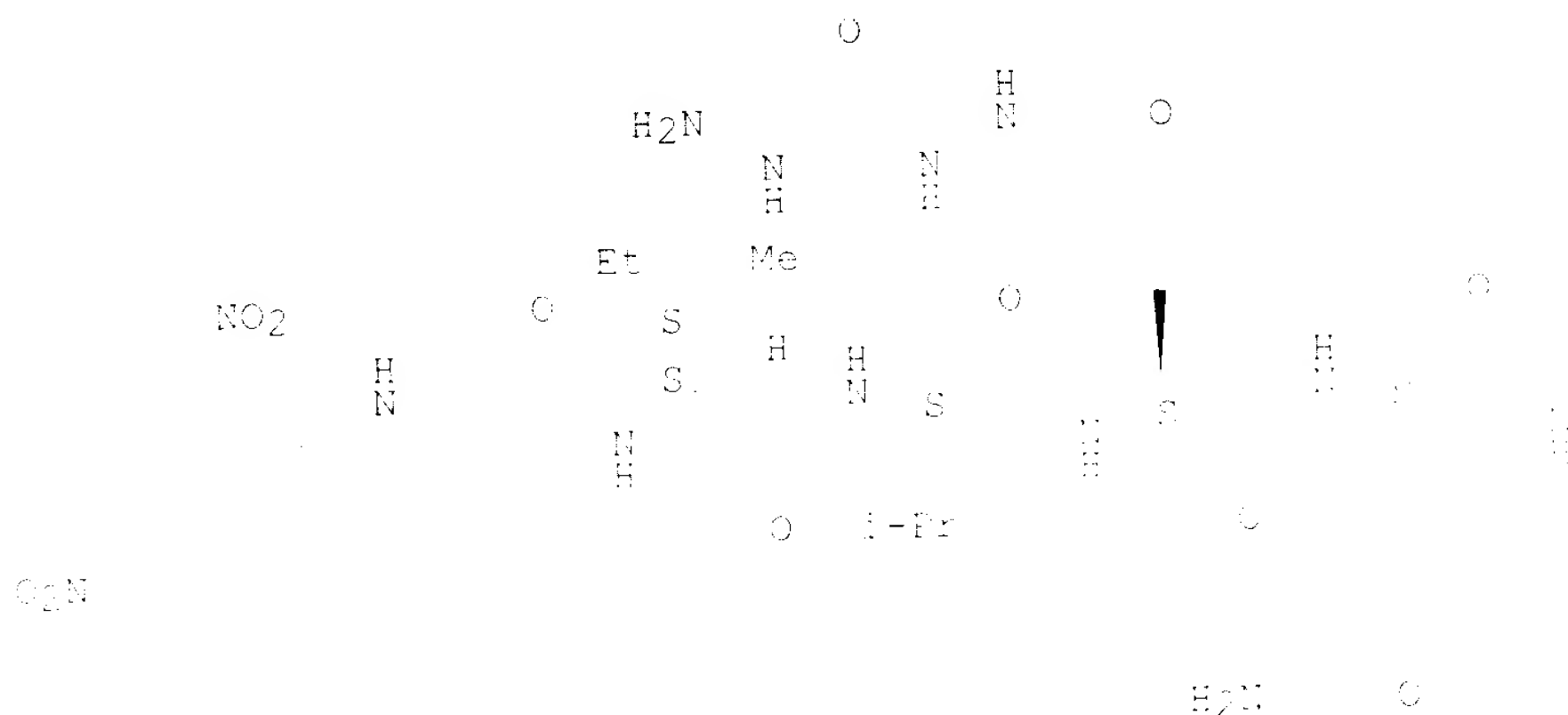


TABLE 1-1

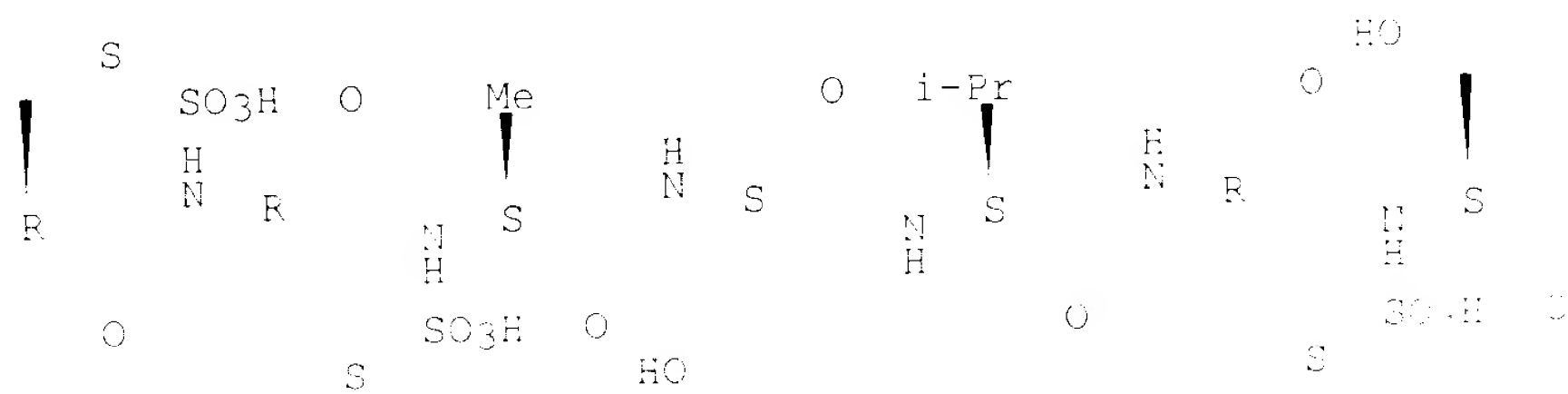
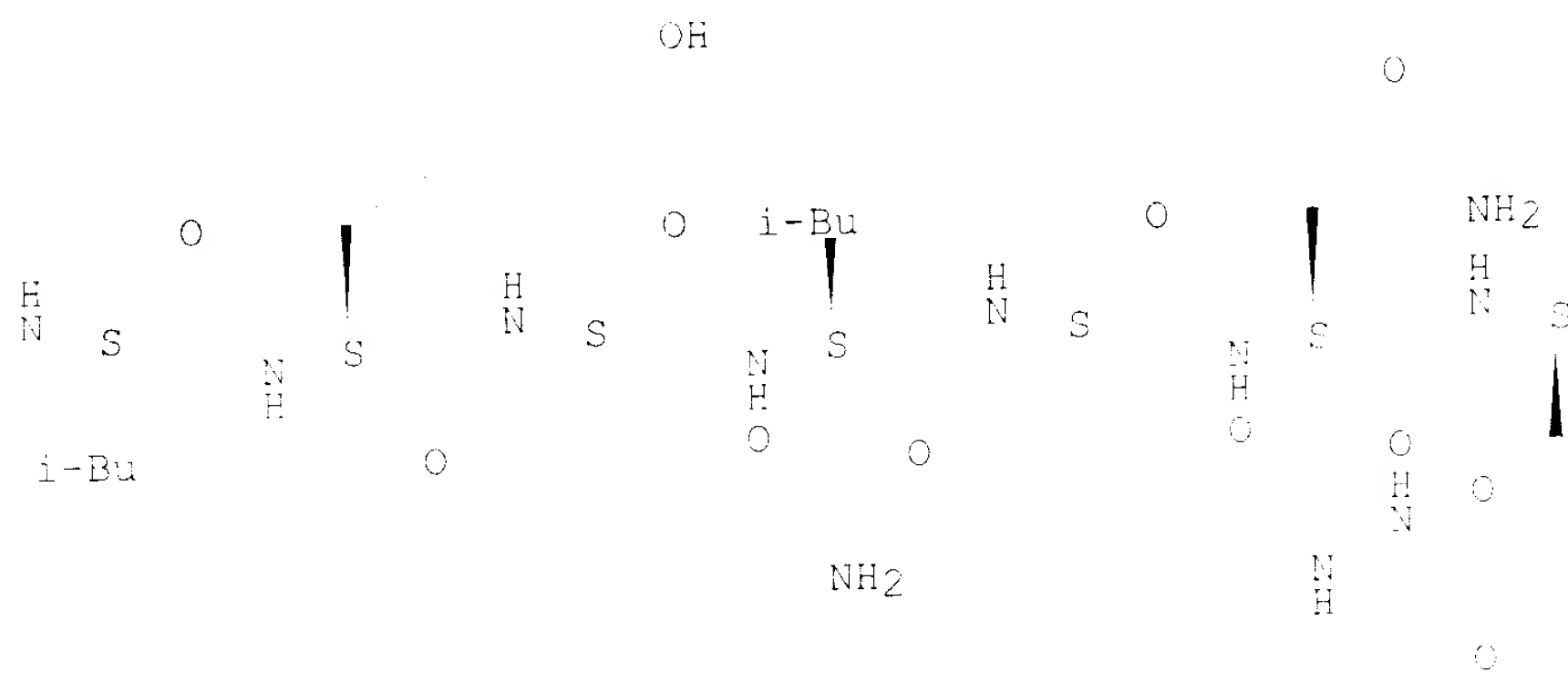
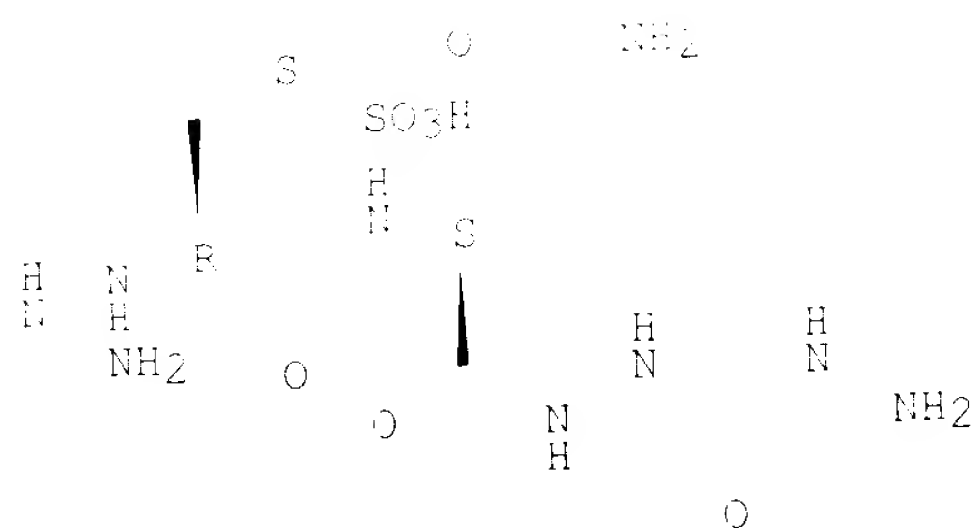


TABLE 1-2



OH



1 REFERENCES IN FILE CA (1962 TO DATE)
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 115:25407

L20 ANSWER 8 OF 16 REGISTRY COPYRIGHT 2002 ACS
RN **127381-73-1** REGISTRY
CN Hexanoic acid, 6-[(1,3-dihydro-1,3-dioxo-2H-isoindol-2-yl)oxy]-6-oxo-,
2-(triphenylmethyl)hydrazide (9CI) (CA INDEX NAME)
MF C33 H29 N3 O5
SR CA
LC STN Files: CA, CAPLUS, TOXCENTER

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

1 REFERENCES IN FILE CA (1962 TO DATE)
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 113:2671

L20 ANSWER 9 OF 16 REGISTRY COPYRIGHT 2002 ACS
RN **89715-26-4** REGISTRY
CN Pyruvic acid, azine with S-methyl thiocarbamate (7CI) (CA INDEX NAME)
PS 3D CONCORD
MF C5 H10 N4 O2 S
LC STN Files: BEILSTEIN*, CA, CAOLD, CAPLUS, CASREACT
(*File contains numerically searchable property data)

SM-

H N C NH NH₂

Me C CO₂H

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

- 1 REFERENCES IN FILE CA (1962 TO DATE)
- 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)
- 1 REFERENCES IN FILE CACLD (PRIOR TO 1962)

REFERENCE 1: 61:69149

L20 ANSWER 10 OF 16 REGISTRY COPYRIGHT 2002 ACS
 RN 50883-75-5 REGISTRY
 CN Carbonic dihydrazide, (1-methyl-2-oxopropylidene)- (CA INDEX NAME)
 OTHER NAMES:
 CN (α-Acetylenehydrazide)carbohydrazide
 FS 3D CONCORD
 MF C5 H10 N4 O2
 LC STN Files: BEILSTEIN*, CA, CAPLUS, TOXCENTER
 (*File contains numerically searchable property data)

O

H₂N NH C NH N O

Me C C Me

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

- 1 REFERENCES IN FILE CA (1962 TO DATE)
- 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 80:48710

L20 ANSWER 11 OF 16 REGISTRY COPYRIGHT 2002 ACS
 RN 14994-19-5 REGISTRY
 CN Carbamoyl azide, terephthaloyldi- (8C1) (CA INDEX NAME)
 FS 3D CONCORD
 MF C10 H6 N8 O4
 LC STN Files: CA, CAPLUS

C O

C NH C N3

N3 C NH C

O O

- 1 REFERENCES IN FILE CA (1962 TO DATE)
- 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 66:05607

L19 ANSWER 12 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 14381-17-0 REGISTRY

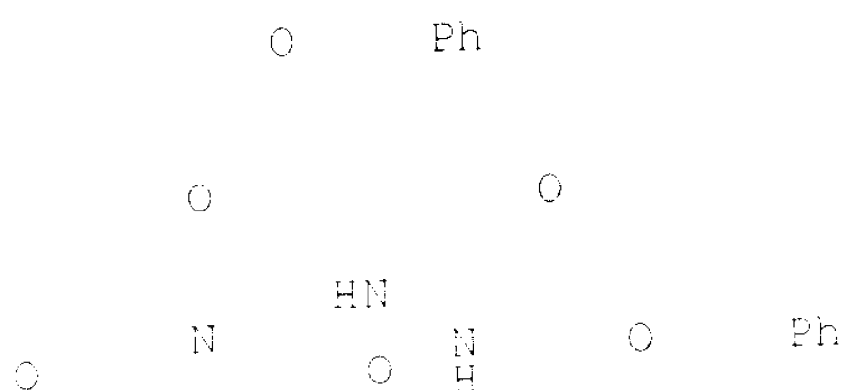
CN Succinimide, N-[[α -(2-carboxyhydrazino)hydrocinnamoyl]oxy]-, tert-butyl ester, DL- (8CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Hydrocinnamic acid, α -(2-carboxyhydrazino)-, α -tert-butyl ester, O-succinimido deriv., DL-

MF C21 H21 N3 O6

LC STN Files: BEILSTEIN*, CA, CAPLUS
(*File contains numerically searchable property data)



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

2 REFERENCES IN FILE CA (1962 TO DATE)
2 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 67:117258

REFERENCE 2: 66:55728

L20 ANSWER 13 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 14381-16-9 REGISTRY

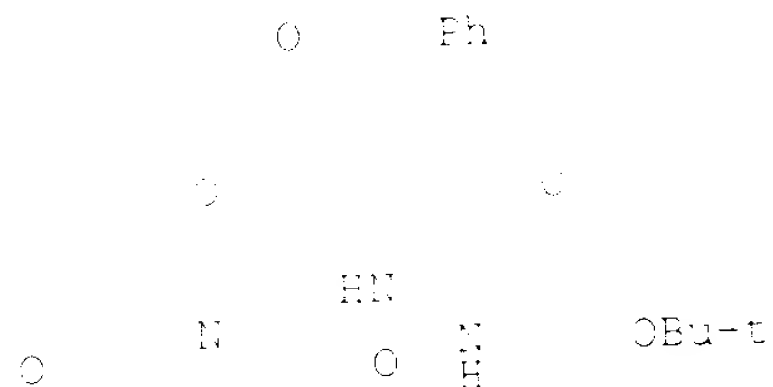
CN Succinimide, N-[[α -(2-carboxyhydrazino)hydrocinnamoyl]oxy]-, tert-butyl ester (8CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Hydrocinnamic acid, α -(2-carboxyhydrazino)-, α -tert-butyl ester, O-succinimido deriv., DL-

MF C15 H23 N3 O6

LC STN Files: BEILSTEIN*, CA, CAPLUS
(*File contains numerically searchable property data)



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

RUSSEL 03 / -10474

1 REFERENCED IN FILE CA (1962 TO DATE)
1 REFERENCED IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 66:15028

120 ANSWER 14 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 13506-12-2 REGISTRY

CN Semicarbazide, 4,4'-phthaloylbis[1-phenyl- (8CI) (CA INDEX NAME)

OTHER NAMES:

CN Carbamic acid, terephthaloyldi-, bis(2-phenylhydrazide)

FS 3D CONCORD

MF C22 H20 N6 O4

LC STN Files: BEILSTEIN*, CA, CAPLUS

(*File contains numerically searchable property data)

O O

C NH C NH NHNH

PhNH NH C NH C

O O

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

2 REFERENCES IN FILE CA (1962 TO DATE)

2 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 69:52073

REFERENCE 2: 66:75807

120 ANSWER 15 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 2509-12-8 REGISTRY

CN Acetic acid, (carbonothioylidihydrazinylylidene)tetra-, tetramethyl ester
(8CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Acetic acid, [(thiocarbonyl)dihydrazinylylidene]tetra-, tetramethyl ester
(7CI)

FS 3D CONCORD

MF C13 H22 N4 O3 S

LC STN Files: BEILSTEIN*, CA, CACLD, CAPLUS

(*File contains numerically searchable property data)

O

S CH2 C OMe

C NH C NH N CH2 C OMe

MeO C CH2 N CH2 C OMe O

O

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

RUSSEL 09 / 815976

2 REFERENCES IN FILE CA (1962 TO DATE)
2 REFERENCES IN FILE CAPLUS (1962 TO DATE)
1 REFERENCE IN FILE CAOLD (PRIOR TO 1962)

REFERENCE 1: 63:38639

REFERENCE 2: 63:38638

L20 ANSWER 16 OF 16 REGISTRY COPYRIGHT 2002 ACE

RN 2215-00-1 REGISTRY

CN Acetic acid, 2,2',2'',2'''-(carbonothioyl-di-2-hydrazinylyl-ylidene)tetrakis- (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CH Acetic acid, [(thiocarbonyl)dihydrazinylylidene]tetra- (7CI, 8CI)

OTHER NAMES:

CN 1,5-Thiocarbohydrazidotetracetic acid

FS 3D CONCORE

MF C9 H14 N4 O8 S

LC STN Files: BEILSTEIN*, CA, CAOLD, CAPLUS

(*File contains numerically searchable property data.)

S CH2 CO2H

NH C NH N CH2 CO2H

HO2C CH2 N CH2 CO2H

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

3 REFERENCES IN FILE CA (1962 TO DATE)
3 REFERENCES IN FILE CAPLUS (1962 TO DATE)
1 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 78:158585

REFERENCE 2: 72:8987

REFERENCE 3: 63:38638